PB82-103623

Small Size-Range Extension of an Optical Particle Counter

Colorado School of Mines
Golden

Prepared for

Environmental Sciences Research Lab.
Research Triangle Park, NC

Aug 81

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SMALL SIZE-RANGE EXTENSION OF AN OPTICAL PARTICLE COUNTER

by

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CR 807089

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TECHNICAL REPORT DATA
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1. REPORT NO. EPA-600/2-81-144	2.
3. RECIPIENT'S ACCESSION NO. PB82 103623	
4. TITLE AND SUBTITLE SMALL SIZE-RANGE EXTENSION OF AN OPTICAL PARTICLE COUNTER	5. REPORT DATE August 1981
	6. PERFORMING ORGANIZATION CODE U253063
7. AUTHOR(S) James T. Brown, Jr.	8. PERFORMING ORGANIZATION REPORT NO.
9. PERFORMING ORGANIZATION NAME AND ADDRESS Colorado School of Mines Golden, CO 80401	10. PROGRAM ELEMENT NO. CAFA1D/01-1164 (FY-81)
	11. CONTRACT/GRANT NO. R807089
12. SPONSORING AGENCY NAME AND ADDRESS Environmental Sciences Research Laboratory - RTP, NC Office of Research and Development U.S. Environmental Protection Agency Research Triangle Park, N. C. 27711	13. TYPE OF REPORT AND PERIOD COVERED FINAL 10/79 - 3/81
	14. SPONSORING AGENCY CODE EPA/600/09

15. SUPPLEMENTARY NOTES

16. ABSTRACT

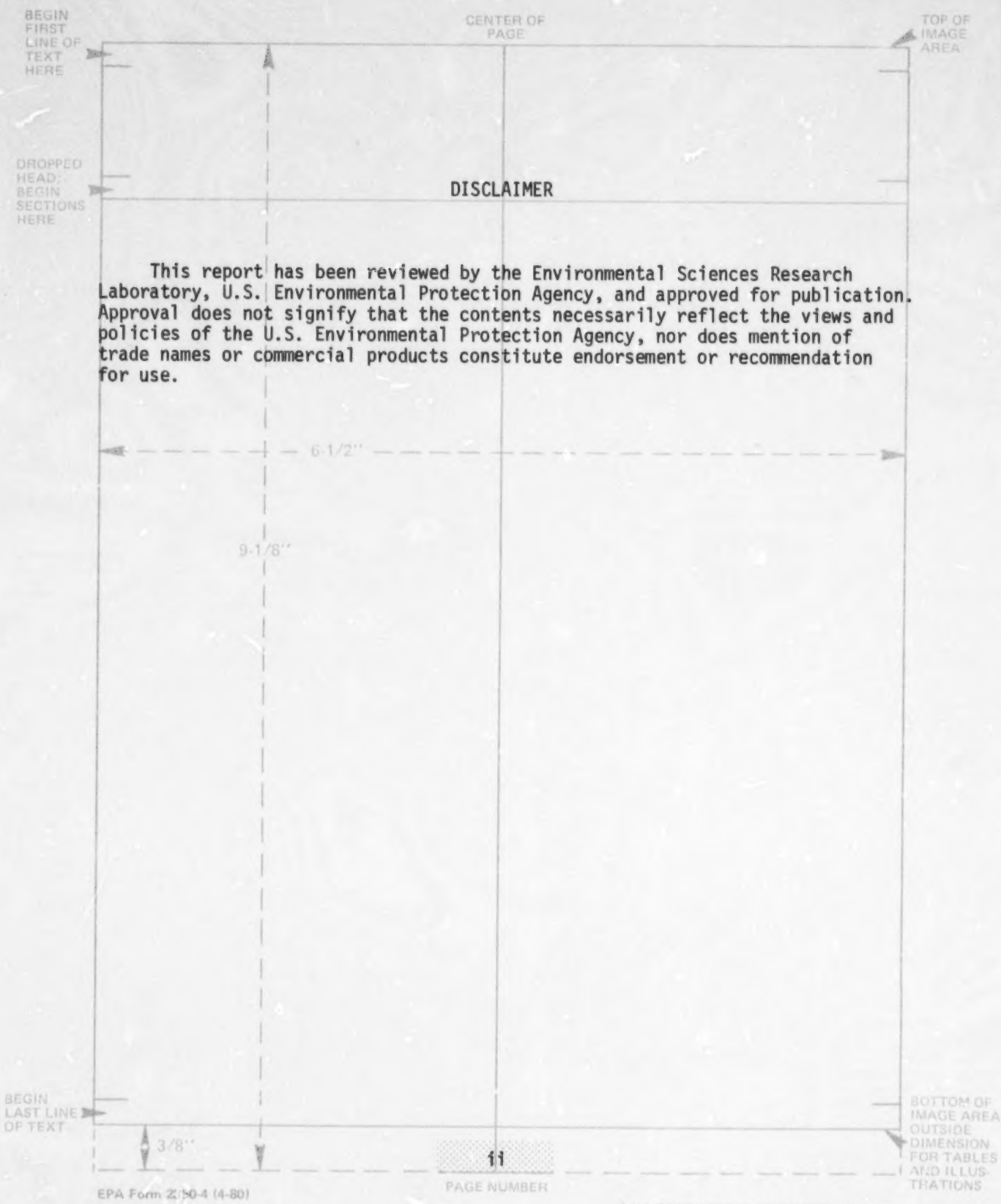
The object of the study was to improve the small particle sensitivity and resolution of a white light optical particle counter. The particular counter chosen for study was the Model 208 manufactured by Climet Instruments. The investigation involved three distinct approaches: (1) a comparison of the effect of several different photomultiplier detectors and operating parameters, (2) the effect of higher temperature operation of the white light source, and (3) modification of the detector and scattering chamber geometries. Of the three possibilities for performance improvement the last was found to be the most useful.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group

18. DISTRIBUTION STATEMENT RELEASE TO PUBLIC	19. SECURITY CLASS *(This Report)* UNCLASSIFIED	21. NO. OF PAGES 20
	20. SECURITY CLASS *(This page)* UNCLASSIFIED	22. PRICE

EPA Form 2220-1 (Rev. 4-77) PREVIOUS EDITION IS OBSOLETE



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ABSTRACT

The object of the study described in this report was to improve the small-particle sensitivity and resolution of a white light optical particle counter. It was expected that the goals of the project could be attained through a series of modifications to the instrument, a Climet 208. First the response of the system was studied as a function of several different photomultiplier (PMT) detectors and operating parameters. Second, the dependence of the sensitivity and resolution on the incident light intensity was examined. Finally, the geometry of the detector and scattering chamber was modified to minimize noise and to improve the sub-micron resolution of the entire system.

The studies of the various PMTs and the anode high voltage have indicated that there is an optimal gain at which to operate each PMT. Contrary to initial expectations, the performance of the RCA 4832 which was selected because of its uniform response characteristic from 300 nm to 900 nm, was disappointing. The RCA 931B usually supplied with the Climet 208 operated near 1000 V. appeared to provide the best small particle response.

As regards the operating conditions of the white light source, a 45W. quartz-halogen lamp, it was found that the higher the effective temperature of the filament the better the small particle size resolution. Operating the quartz-halogen bulbs at elevated temperature (increased filament current), however, shortens the lifetime of the lamp. Forced air circulation around the lamp is recommended for these more extreme conditions.

In the last phase of the study, the geometry of the scattering chamber was slightly modified to permit a refocusing of the scattered light (signal) on the PMT in such a way as to minimize the size of the image of the scattering volume on the surface of the PMT. This refocusing then permits masking of the PMT which significantly reduces the stray scattered light that reaches the photocathode. The quasi-quantitative results for the mask size, approximately 4.5 mm. diameter, agrees well with the aperture calculation based on a consideration of only axial rays originating in the scattering volume.

This report was submitted in fulfillment of Cooperative Agreement No. CR807089 by the Colorado School of Mines under the sponsorship of the U.S. Environmental Protection Agency. This report covers the period October 1, 1979 to March 1, 1981, and work was completed as of March 31, 1981.

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ACKNOWLEDGEMENT

The contributions of the EPA project officer, Dr. Charles W. Lewis, to the results of this investigation are gratefully acknowledged. His suggestion of the use of photographic film for image characterization was particularly useful.

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INTRODUCTION

In many applications the characteristics of single particle optical counters are desirable because they are able to provide both size and number information on a near real-time basis. Typically the operating (size) range for these instruments extends from tens of microns down to several tenths of a micron. (Particle size refers to geometrical diameter throughout this report). Because the physiological impact of particles as small as 0.1 micron may be significant, it would be advantageous to be able to apply single particle optical detectors to problems and research in this size range. A general review of light scattering theory as applied to optical counters has been given by Hodkinson (1966).

The instrument chosen for this study is the Climet 208 (Climet Instruments Co., Redlands, CA); its nominal size limit is 0.3 to 10 microns. In practice it is difficult to resolve distributions for which the mean size is less than 0.5 micron. (See for example the data reported by Lewis and Lamothe (1978) for a standard Climet 208). Familiarity with the Climet 208 has indicated several potential modifications that show promise for improving its resolution below 0.5 micron and increasing its sensitivity for particles as small as 0.1 micron. The increased sensitivity was expected to result primarily from decreases in the noise intrinsic to the design of the system. The improvements in resolution were predicted to result from size-dependent gain characteristics including the photomultiplier tube (PMT) spectral response and the spectral distribution of incident light.

In order to obtain a relatively unambiguous evaluation of the sub-0.5 micron response of this instrument it was necessary to have access to an aerosol with a well defined peak in its size distribution. Of equivalent importance is the absence of a large background of smaller particulates. A parallel-plate electrostatic classifier of recent design was available and could provide the required monodisperse aerosol by separating the off-sized residuals from an aerosol generated from Dow latex spheres (Dow Diagnostics, Indianapolis, IN). The refractive index of such particles (at a wavelength of 0.59 micron) is 1.59, which is similar to values commonly assumed for ambient aerosol.

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CONCLUSIONS AND RECOMMENDATIONS

The results of this project have been very encouraging. It has been demonstrated that it is possible to extend the small-size sensitivity of the Climet 208 to allow resolution of a 0.312 micron aerosol peak. The most significant modifications in the instrument leading to this improvement are the refocusing of the elliptical mirror on the PMT, the masking of the surface of the PMT, and increasing the operating temperature of the quartz-halogen lamp.

The anticipated improvement in the response through the use of a broad spectral response PMT (RCA 4832) did not occur. Lack of resources, however, limited the thoroughness of this part of the investigation, and additional work is recommended to understand and confirm this result.

It is expected that a more lengthy and detailed study of additional PMT characteristics and improved flow control for hydrodynamic focusing of the particle laden air stream would lead to additional resolution improvements. Also, since this study had as its intent the improved response for particles in the 0.1 to 0.5 micron range, the influence of the proposed modifications and operating parameters will narrow the applicability of this instrument in size ranges of interest for other applications.

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DESCRIPTION OF THE OPTICAL PARTICLE COUNTER (Climet 208)

Figure 1 presents a schematic view of the Climet 208 sensing head. Light from a 3000°K quartz-halogen lamp is scattered from a collimated beam. The scattered light is collected by an elliptical mirror and focused on a PMT detector. The scattering volume, which is the intersection of the particle beam with the collimated light beam, is placed at one focus of the elliptical mirror and the PMT is placed near the other focus. In the application under consideration the electronics which are supplied by the manufacturer are bypassed and the output of the PMT is fed directly to an amplifier and a multichannel analyzer (MCA).

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In order to have greater flexibility in the gain of the PMT, the high voltage power supply provided with the instrument was replaced with a separate high voltage power supply. Similarly the D.C. power supply for the quartz-halogen lamp was replaced so that the lamp current, and consequently the filament temperature and spectral composition of the incident light, could be varied and carefully regulated.

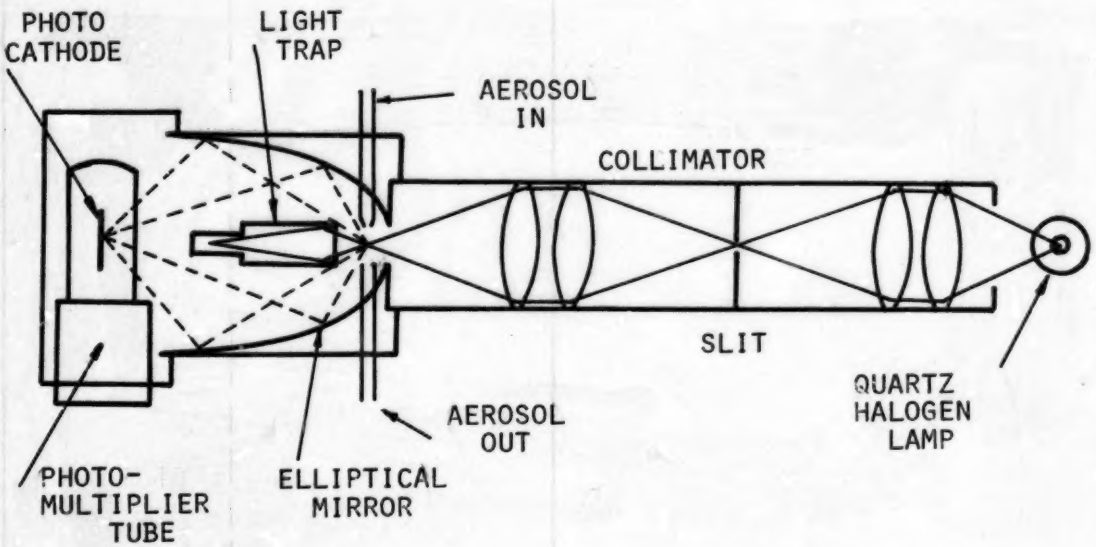

Figure 1. Climet 208 sensing head

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TEST AEROSOL PREPARATION - ELECTROSTATIC CLASSIFIER

In order to evaluate the response of a particle counter, it is essential to have a source of relatively uniform sized particles. The more common choices for calibration aerosols often include those produced by aerosol generators and commercially prepared suspensions of latex spheres.

A well known shortcoming of producing calibration aerosols from latex suspensions is the unavoidable production of off-sized particulates in resuspending the latex spheres. The instances in which two or more latex spheres (multiplets) are dispersed in the same droplet can be controlled by diluting the suspension. Additionally, these multiplets do not interfere significantly with efforts to improve the small-size resolution because they always appear as larger in size than the principal peak of single latex spheres. The most difficult aspect of using redispersed latex spheres for studies of the nature of those described in this report is that an overwhelming number of extremely small (less than 0.3 microns) residual particulates are produced. Most of these residuals result from the evaporation of a droplet and contain only the emulsifier and no latex sphere. In order to evaluate the sub-0.5 micron response of the particle counter, these residuals must be removed.

Fortunately there are methods which are capable of classifying particles according to their size. In this case a parallel-plate electrostatic classifier developed by Schowengerdt and Brown (1980) was available. An electrostatic classifier separates particles according to their electrical mobility which is inversely proportional to the particle diameter.

In order to remove the background of residuals, the aerosol was first brought to charge equilibrium by passing it through a commercially available krypton (85) source. The radioactive krypton source brings the aerosol to bipolar charge equilibrium after which it is injected in a sheath of filtered air between two oppositely charged parallel plates as in Figure 2. Because of the inverse size dependence of the electrical mobility, the smaller particles are readily deflected to the metal plates whereas the larger particles travel further along the space between the plates before being impacted on them. The classifier has a slit cut in each plate so that the particles following a certain trajectory can be removed from between the plates. Since the trajectory is size dependent, the flows and electric field can be adjusted to cause a desired size particle to emerge through the slits and be extracted for calibration purposes. In the above fashion a large fraction of the off-sized particles (both large and small) can be removed from the sample which may then be used for the calibration of the optical particle counter.

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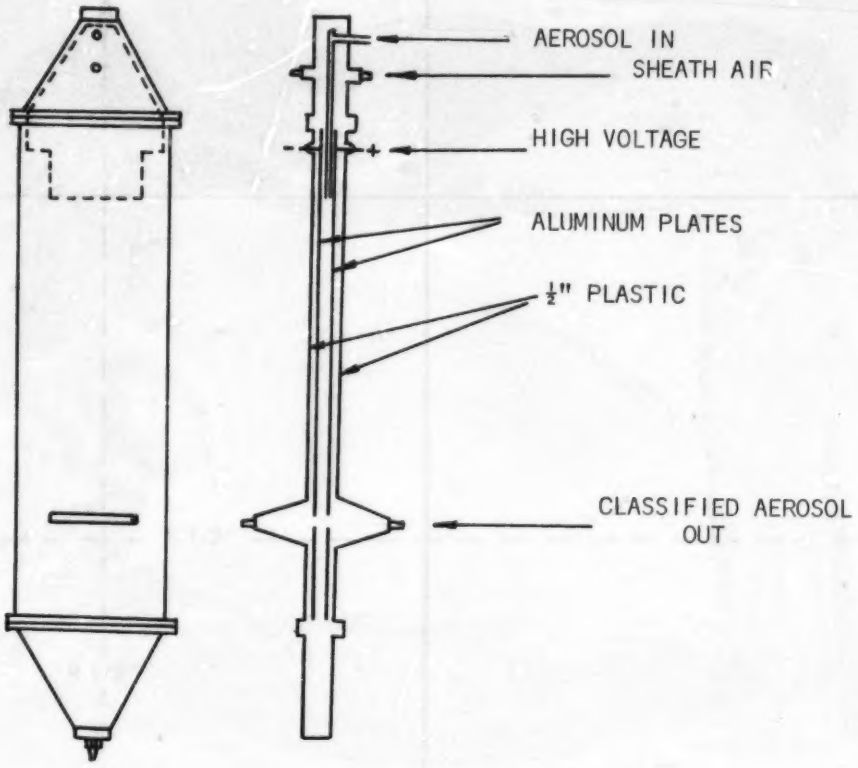

Figure 2. Parallel-plate electrostatic particle classifier

PHOTOMULTIPLIER EVALUATION

The objectives of this aspect of the investigation were to evaluate the advantages of a higher gain PMT and to determine what improvements were to be had through the application of a broad spectral response PMT. The PMT supplied with the Climet 208 was the RCA 931A and in some more recent models, the RCA 931B. Each of these PMTs is characterized by maximum sensitivity in the blue part of the spectrum, the primary difference between them being that the gain of the 931B is approximately 4 times greater than that of the 931A. It was expected that a PMT with photocathode characteristics such as those for GaAs in the RCA 4832 might be well suited for the detection of light scattered by the small particles of interest here. Figure 3 shows the relative responsivities for the PMTs under consideration. As can be seen, for the 4832 the responsivity is quite large and relatively flat from 300 nm to beyond 800 nm. Since the smaller particles "qualitatively" scatter the shorter wavelengths more efficiently, the higher responsivity for the shorter wavelength was expected to effect an overall improvement.

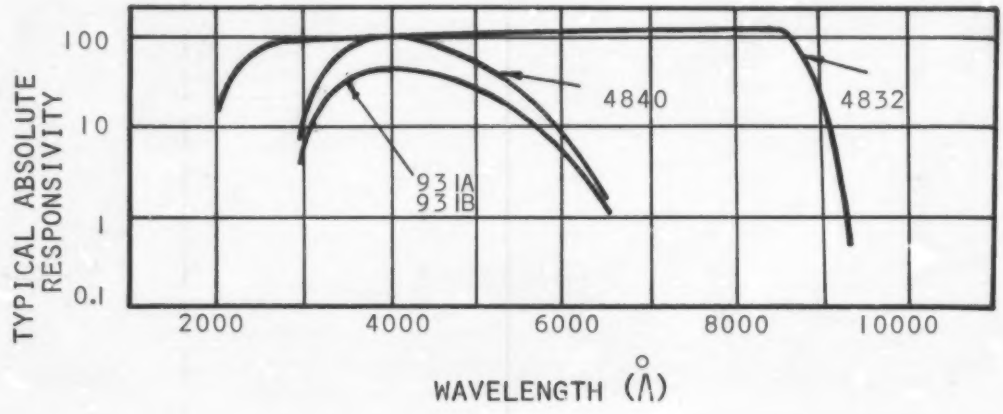

Figure 3. Photomultiplier spectral response curves

The evaluation of the various PMTs was on a somewhat subjective basis. First, the intrinsic noise level as a function of the high voltage was measured. As a result of this procedure, where only filtered air was passed through the aerosol inlet, the major shortcoming of the RCA 4832 became apparent; the noise level showed a rather sharp increase with increasing anode voltage. The noise level in the RCA 4832 was found to be approximately four

times the level for the noise in the 931B. Additional study would be necessary to specify the sources of the noise associated with the use of the 4832 or any of the PMTs included here. Similar measurements on the 931B indicated that 1000 V. was optimal. Compared to the 931B, the 931A showed comparable characteristics but the aforementioned lower gain makes its use a little more awkward in the application under consideration. The RCA 4840 certainly merits consideration on the basis of its specifications; this type of tube was not available in time to be included in this report.

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LIGHT SOURCE EVALUATION

The standard white light source provided with the Climet 208 is a 45 watt, 6.6 ampere, quartz-halogen lamp. The effective filament temperature for the standard operating conditions is about 3000°K. The rationale behind light source modifications was primarily that it would be desirable to increase the operating temperature of the filament because of the increased output in the short wavelength portion of the spectrum. The results of operating the quartz-halogen lamp at elevated temperatures can be seen in Figure 4. (The original data for Figures 4 and 6 are photographs from the oscillographic display of the MCA. Because of the poor quality of the photos, tracings are presented here.) The five spectra were obtained using 0.460 micron latex spheres and by varying the lamp voltage from 6.0 V. to 10.0 V. in steps of 1.0 V. In Figure 4, some spectra occur with different vertical scales, however, this does not influence the conclusion that the resolution is substantially improved with increasing lamp voltage and filament temperature. The expected steady progression of the peak position with increasing lamp voltage is also apparent. Since increasing the filament temperature will significantly reduce the expected lifetime of the bulb, any decision to routinely operate the system under such conditions will necessarily have to consider the inconvenience of frequent bulb replacement; at the maximum voltage, the bulb lifetime is expected to be of the order of several to tens of hours. Bulb-life as a function of operating voltage was not studied quantitatively.

An additional modification to the light source has been included; a cylindrical mirror with the filament of the lamp along the axis. This mirror has the property of focusing about 20 percent of the total light emitted by the bulb (away from the collimator) back on the filament. It is estimated that the mirror increases the effective emissivity of the filament by 10 to 20 percent. Because the mirror is quite awkward to remove, its effect has not yet been evaluated but does not appear significant. Perhaps a much more compelling reason for including such a mirror, which is made of polished aluminum tubing, is that it provides additional protection from shattering of the quartz-halogen lamp which can occur under the more extreme operating conditions described here.

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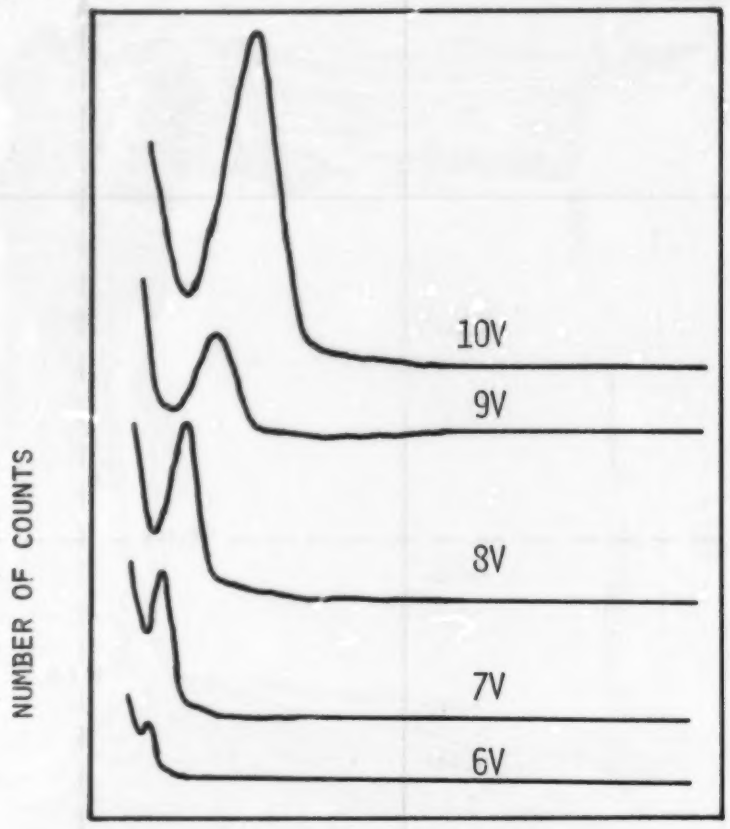

Figure 4. Particle size spectrum as a function of lamp voltage for 0.46 micron latex spheres. The vertical scale is arbitrary for each spectrum.

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MODIFICATION OF THE DETECTOR

The single most important source of noise that leads to the response degradation of the Climet 208 is spurious scattered light. The contributions to the spurious light signals include: Rayleigh scattering along the incident light beam, imperfections and dust in the light trap, residual suspended particulates passing through the incident light beam outside the scattering volume, diffraction, and dirty optical components. The scattering volume is the intersection of the particle beam and the incident light beam where the slit is imaged. The diameter of the particle beam is 1.1 mm and the thickness of the image of the slit at the focus is 0.7 mm.

Since the extent of the scattering volume is so small, it is clear that virtually all sources of noise lie outside of it; this observation leads to the suggestion that the elliptical mirror may "focus" the light from various points within the detector at points on the photocathode different from the image of the scattering volume on the photocathode. Consequently, the focal properties of the elliptical mirror were examined. First, a piece of photographic paper was fastened to the surface of the PMT and a .005 inch wire was threaded through the inlet and outlet ports intersecting the incident light beam. The wire then served as a source of scattered light originating in the scattering volume. Exposing and developing the film yields the desired image information. Calculations based on the geometry and the mirror configuration agree very well with the image determination just described. In each case, the image of the scattering volume is found to be a 5 mm diameter circle at the focal point of the mirror. Since the dimensions of the photocathode are large (8 mm x 24 mm), it should be possible to mask the surface of the PMT to admit only the signals falling on the image of the scattering volume. In an effort to estimate the extent of the image of the noise, the illuminated portion of the photocathode due to light scattered from regions outside of the scattering volume as described above, the wire was removed and the PMT covered again with photographic paper. With no signal, that is, no particulates passing through, a long exposure was made. At the focal point of the mirror the image produced by the intrinsic noise was a circle approximately 20 mm in diameter. Because long exposures were required to define the noise image, the spurious signals must be small and would probably interfere mostly with measurements involving very small particles.

Also, in order to optimally mask the PMT, the sensing head had to be adjusted to place the surface of the PMT at the focal point of the mirror, further from the mirror housing than the normal operating configuration. It was not possible to adjust the Climet 208 to this position because, with the required extension of the sensing head, the o-rings would no longer provide a reliable seal. Figure 5 shows the "collar" attachment that was fabricated to fit onto the outside of the sensing head to allow the additional extension

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needed to focus the mirror on the PMT surface. The masking of the PMT was accomplished by taping a piece of black-painted aluminum foil, with the appropriate circular aperture, onto the glass envelope of the PMT. As can be seen in Figure 6, the improvement in resolution due to the masking is appreciable for 0.3 micron latex aerosols. For large aerosols, the modifications to the focal adjustment and masking the PMT do not appear advantageous. Since the emphasis of this work was a study of the small-size response of the Climet 208, the effects of the modifications described herein on the large particle response has not been examined in detail.

Although not investigated in this study, additional noise reduction is almost certainly possible through modification of the light trap to a "horn" configuration.

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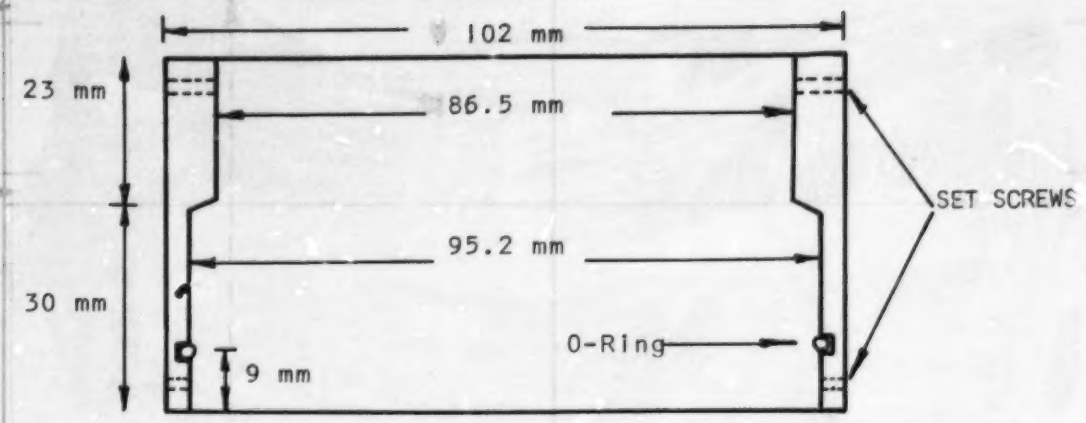

Figure 5. Schematic drawing of sensing head adaptor

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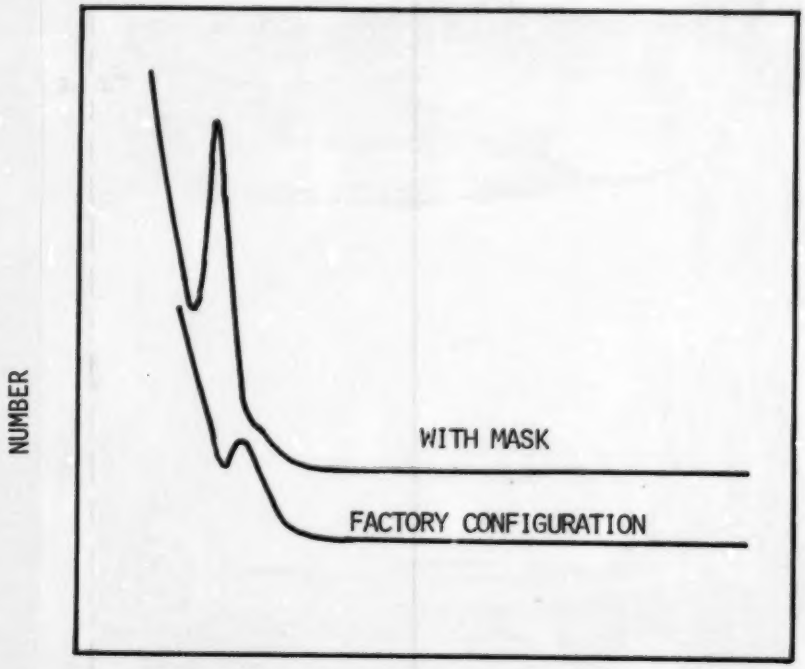

Figure 6. Particle size spectrum for 0.312 micron latex showing the improved resolution due to masking the PMT.

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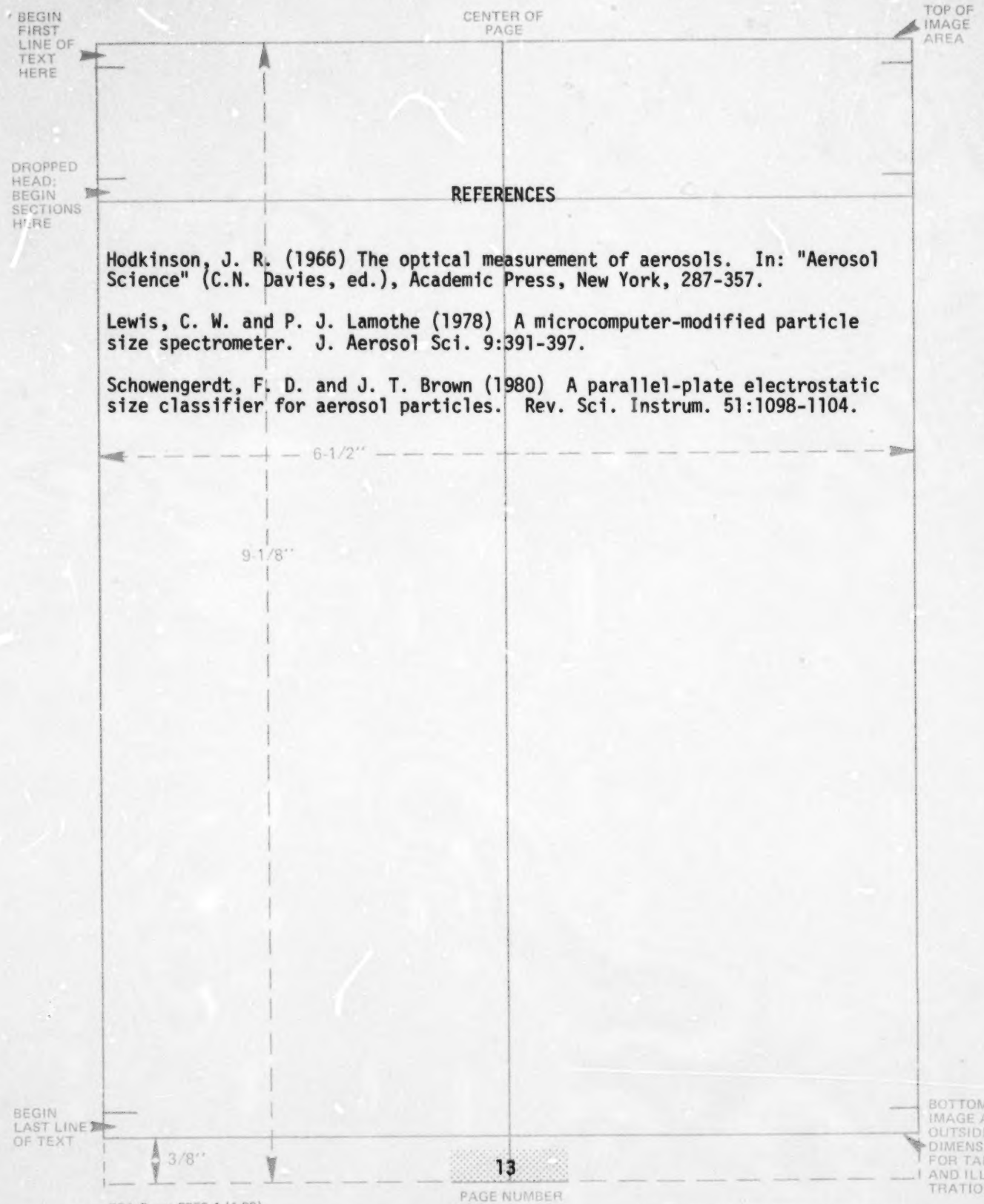
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